

0684

**REPORT DOCUMENTATION PAGE**

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing this collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number. **PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.**

<b>1. REPORT DATE (DD-MM-YYYY)</b> 5/11/04		<b>2. REPORT TYPE</b> annual progress Report		<b>3. DATES COVERED (From - To)</b> Nov 1, 2003 - Oct 31, 2004	
<b>4. TITLE AND SUBTITLE</b> Development of Composites Reinforced with Short Wavy Fibers				<b>5a. CONTRACT NUMBER</b>	
				<b>5b. GRANT NUMBER</b> F49620-02-1-0018	
				<b>5c. PROGRAM ELEMENT NUMBER</b>	
<b>6. AUTHOR(S)</b> C. T. Sun				<b>5d. PROJECT NUMBER</b>	
				<b>5e. TASK NUMBER</b>	
				<b>5f. WORK UNIT NUMBER</b>	
<b>7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)</b> Purdue University West Lafayette, Indiana 47907				<b>8. PERFORMING ORGANIZATION REPORT NUMBER</b>	
<b>9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)</b> Air Force Office of Scientific Research 4015 Wilson Blvd., Rm 713 Arlington, VA 22203-1954 NA				<b>10. SPONSOR/MONITOR'S ACRONYM(S)</b> AFOSR, Dr. Les Lee	
				<b>11. SPONSOR/MONITOR'S REPORT NUMBER(S)</b>	
<b>12. DISTRIBUTION / AVAILABILITY STATEMENT</b> DISTRIBUTION UNLIMITED					
<b>13. SUPPLEMENTARY NOTES</b>					
<b>14. ABSTRACT</b> <p>In short fiber composites including nanocomposites, the load transfer efficiency among fibers is crucial in effecting superior composite properties. It is conceivable that this load transfer efficiency depends on the shape, aspect ratio, and surface area of the fiber. The effect of surface area of the reinforcing element is of particular importance because of the increasing use of nano particles in nanocomposites. It is well known that for the same volume, a material at nano scale possesses much greater surface areas than at larger scales. It is evident that more surface areas mean more load transfer paths and, thus, lower interfacial stresses between the reinforcement and the matrix. The lowering of interfacial stresses is expected to lead to higher composite strengths. In the previous period, we used model composites to reach the following conclusions: 1) Wavy fibers lower the interfacial stresses and thus increase the composite strength significantly, and 2) For the same fiber volume fraction, the composite with thinner fibers has higher strength than the composite with thicker fibers. In this reporting period, we continued to investigate the size effect of reinforcements. In addition to platelets, we also included spherical particulates of varying sizes. For the platelet reinforcement, composites are manufactured with steel platelets whose lateral dimensions are on the order of centimeters and thickness in the range of 0.01 - 1.0 mm. Pull-out tests are conducted and micromechanics models are developed to understand the reinforcing mechanisms and to develop a strength prediction method. For particle reinforcements, glass beads of 2000 <math>\mu\text{m}</math>, 500 <math>\mu\text{m}</math>, 200 <math>\mu\text{m}</math>, 70 <math>\mu\text{m}</math> and 6 <math>\mu\text{m}</math> in diameter (mean size) and alumina particles of 70 <math>\mu\text{m}</math>, 20 <math>\mu\text{m}</math>, 3 <math>\mu\text{m}</math>, and 50 nm in diameter are used together with vinyl ester to make composites for evaluating the size effect on the composite stiffness and strength properties.</p>					
<b>15. SUBJECT TERMS</b>					
<b>16. SECURITY CLASSIFICATION OF:</b>			<b>17. LIMITATION OF ABSTRACT</b>	<b>18. NUMBER OF PAGES</b> 8	<b>19a. NAME OF RESPONSIBLE PERSON</b> C.T. Sun
<b>a. REPORT</b> UNCLASSIFIED	<b>b. ABSTRACT</b> UNCLASSIFIED	<b>c. THIS PAGE</b> UNCLASSIFIED			<b>19b. TELEPHONE NUMBER (include area code)</b> 765-494-5130

20050104 031

**DISTRIBUTION STATEMENT A**  
Approved for Public Release  
Distribution Unlimited

12-27-04

## Introduction

In short fiber composites including nanocomposites, the load transfer efficiency among fibers is crucial in effecting superior composite properties. It is conceivable that this load transfer efficiency depends on the shape, aspect ratio, and surface area of the fiber. In an earlier study, this PI found that a wavy lap joint configuration could yield much greater joint strengths than the conventional flat lap joints [1]. This great improvement in strength of the wavy joint was found to be the result of the interfacial stresses that were altered by the wavy geometry. The effect of surface area of the reinforcing element is of particular importance because of the increasing use of nano particles in nanocomposites. It is well known that for the same volume, a material at nano scale possesses much greater surface areas than at larger scales. It is evident that more surface areas mean more load transfer paths and, thus, lower interfacial stresses between the reinforcement and the matrix. The lowering of interfacial stresses is expected to lead to higher composite strengths. In the previous period, we used model composites to reach the following conclusions: 1) Wavy fibers lower the interfacial stresses and thus increase the composite strength significantly, and 2) For the same fiber volume fraction, the composite with thinner fibers has higher strength than the composite with thicker fibers.

In this reporting period, we continued to investigate the size effect of reinforcements. In addition to platelets, we also included spherical particulates of varying sizes.

## Objective

The objective of this research is to study the efficiency in load transfer in short fibers in composite materials. The variables of the reinforcement such as its shape and surface area/weight ratio will be investigated both theoretically and verified experimentally. It is anticipated that the result of this research will benefit the design of the conventional short fiber composites as well as the emerging nanocomposites in which nano particles have extremely high surface/volume ratios.

## Approach

Platelet and spherical type reinforcements are considered. Attention is focused on the effect of surface area/vol of the reinforcement on the mechanical properties of the composite. For the platelet reinforcement, composites are manufactured with steel platelets whose lateral dimensions are on the order of centimeters and thickness in the range of 0.01 - 1.0 mm. Pull-out tests are conducted and micromechanics models are developed to understand the reinforcing mechanisms and to develop a strength prediction method. For particle reinforcements, glass beads of 2000  $\mu\text{m}$ , 500  $\mu\text{m}$ , 200  $\mu\text{m}$ , 70  $\mu\text{m}$  and 6  $\mu\text{m}$  in diameter (mean size) and alumina particles of 70  $\mu\text{m}$ , 20  $\mu\text{m}$ , 3  $\mu\text{m}$ , and 50 nm in diameter are used together with vinyl ester to make composites for evaluating the size effect on the composite stiffness and strength properties.

## Status of Effort

### Part I: Platelet Reinforcement

The load transfer efficiency in short fiber composites is one of the most crucial factors affecting the overall composite properties. This load transfer efficiency depends on the nature of the fiber namely its shape, size and the surface area. The objective of this part of research is to study the effect of surface area on the efficiency of load transfer in short fiber composites. The effect of surface area on the load transfer efficiency of composites is investigated experimentally and is then verified by finite element analysis.

### Experiment

For easy fabrication and processing, a metal fiber composite system was chosen. The fiber platelet was made using the steel 1095 alloy. Steel sheets of different thickness 0.127mm (0.005 inches), 0.254mm (0.01 inches) and 0.508mm (0.02 inches) were cut into strips 4.6mm wide using a water jet. Vinyl ester matrix was chosen which was made by thoroughly mixing vinyl ester resin with 1.75% (by weight) MEKP hardener. Mold was prepared with the platelet in place and vinyl ester was poured around it producing an 18 mm embedded length in the resin. It was then allowed to cure for 24 hrs at room temperature and then post cured at 250°F for 2 hrs. Once fully cured, the specimen was cut to the desired width. Fig. 1 shows the typical specimen geometry.

Fiber pull-out tests were performed on these specimens. The stress-displacement curves are shown in Fig.2. The sudden drop in the stress values in Fig. 2 represents the point of pull out. The thinner fibers experienced a higher pull out stress than the thicker fibers. As the thickness of the fiber is reduced, the pull out stress increases. There is an increase of about 30% for each reduction in the thickness of the fiber. This increase can be attributed to the fact that by having same volume fraction of fibers and increasing the contact area between the matrix and fibers, there is a better and more efficient stress transfer from fiber to matrix and vice versa

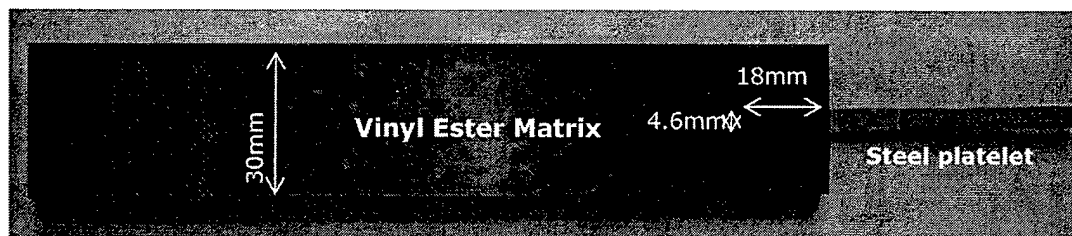


Figure 1: Specimen details

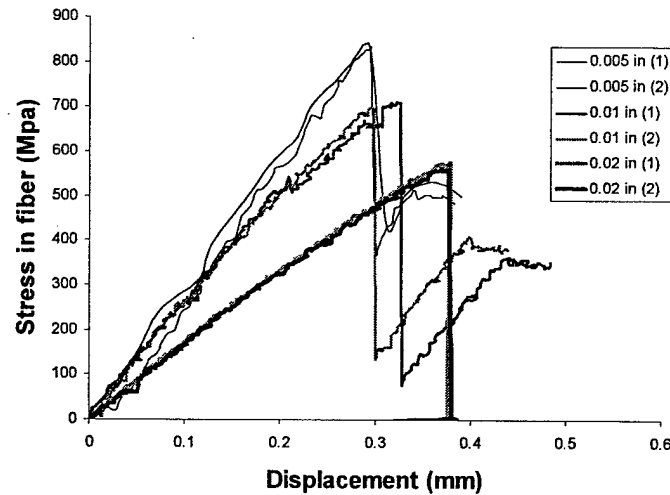


Figure 2: Stress-Displacement curve for fiber pull out tests

### *Finite Element Analysis*

To understand better the effect of thickness on the stress transfer efficiency of short fiber composites, finite element analyses were performed by modeling the thick (0.508mm or 0.02in) and thin fiber (0.254mm or 0.01in) composite specimens in the commercial finite element code ABAQUS. A two-dimensional finite element model as shown in Fig. 4 was constructed. Proper boundary conditions were applied and the fiber was pulled by a uniform stress of 1 MPa. The thickness of the resin was set as 6.5mm and the embedded length of the fiber was 18mm. Linear plane strain elements (Q4-4 noded linear quadrilateral) were used for the analysis. The elastic modulus and Poisson's ratio for the fiber and the matrix was taken to be 165GPa, 3.6GPa and 0.3, 0.36 respectively.

Fig. 3 shows the comparison of the interfacial shear stress distributions in thin and thick fiber specimens. The interfacial shear stress concentration in the thin fiber composite is observed to be less severe than the thick fiber composite. These interfacial stresses near the embedded end for the thicker fiber are significantly greater than those for the thin fiber. From the result of the finite element analysis, it can be concluded that on account of the lower interfacial shear stress distribution by having the same fiber volume fraction, the thin fiber composite has a greater surface area for stress transfer and hence outperforms the thick fiber composite.

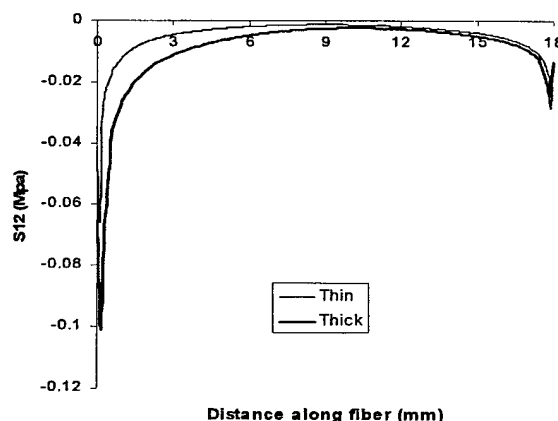


Figure 3: Interfacial shear stresses

## Part II Particle Reinforcement

It is a common practice to add high modulus particles of various sizes in polymers to improve their mechanical properties. The mechanical properties of such composites are mainly dependent on the volume fraction, the size of the particle, and the interfacial behavior between particles and polymeric matrix. In this part of the study, the effects of particle size on the failure mechanism and mechanical performance of the composite are investigated through tensile tests and microscopic observations.

### Experiment

A general purpose vinyl ester was employed as a matrix material. For particles as inclusions, glass beads and aluminum oxide spherical particles were selected. Mean sizes of glass beads were 2000  $\mu\text{m}$ , 500  $\mu\text{m}$ , 200  $\mu\text{m}$ , 70  $\mu\text{m}$  and 6  $\mu\text{m}$  in diameter. For aluminum oxides, mean particle sizes were 70  $\mu\text{m}$ , 20  $\mu\text{m}$ , 3  $\mu\text{m}$ , and 50 nm in diameter.

Unlike the well separated micron sized particles, nanoparticles tend to stick together and form aggregates and agglomerates due to hydrophilic envelope around each particle. Aggregates are usually broken down to agglomerates by mechanical agitation such as shear mixing. Agglomerates, which have edge to edge or point to point contact of particles, can be taken apart and dispersed sonication. However, without a dispersing additive, the particles still tend to reaggregate due to the hydrophilic envelope. A dispersant, BYK-966 manufactured by BYK-Chemie, was added by 5 wt.% of the particles and mixed for 2 minutes with the prepared resin. Based on the designed volume fractions, aluminum oxide nanoparticles were mixed with the resin. As a mechanical agitation, blending was performed for 5 minutes. Subsequently, sonication with a sonicator made by Misonix was applied to the mixture for 20 minutes.

### Results

Figures 4 and 5 show the effect of particle size on Young's modulus and failure stress under tensile loading, respectively. Both properties are normalized with the neat

resin properties. From Fig. 4, it is clear that Young's modulus is dependent on fiber volume fraction of the particles rather than their sizes. However, it is clearly seen in Figure 5 that the failure stress of the composite is significantly dependent on the particle size in the composite, and that strength increases as particle size decreases.

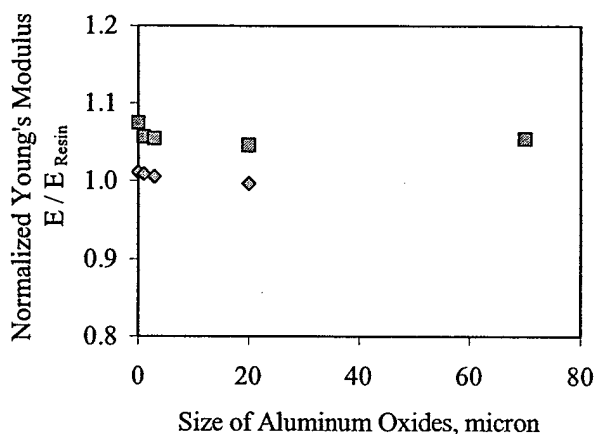


Figure 4 Effect of Particle Size on Young's Modulus with 1 vol. % (3.6 wt.%: 50 nm, 3.7 wt.%: 3–70  $\mu$ m) and 3 vol.% (10.8 wt.%: 50 nm, 11.2 wt.%: 3–70  $\mu$ m) Aluminum Oxides. (Square and diamond symbols indicate normalized Young's modulus with 3 vol. % and 1 vol. % particle loadings, respectively.)

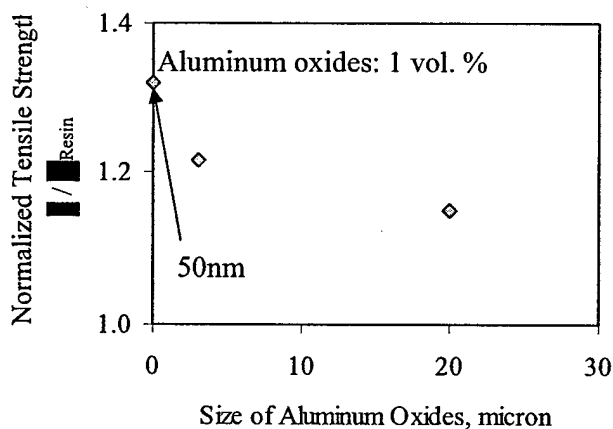


Figure 5 Effect of particle size on tensile strength with 1 vol. % aluminum oxide particles.

In order to observe the failure progression in the composite with micron sized particles, a small load frame as shown in Fig.6 was used to perform tension tests under a microscope. Fig. 7 shows the micrograph at about 92% of the failure load of the composite with 3 vol. % of 20  $\mu$ m aluminum oxide particles. The observed failure

initiation mode was debonding between the particle and the matrix. The subsequent mode of failure is matrix cracking induced by particle/matrix interfacial debonding, which leads to the final failure of the composite.

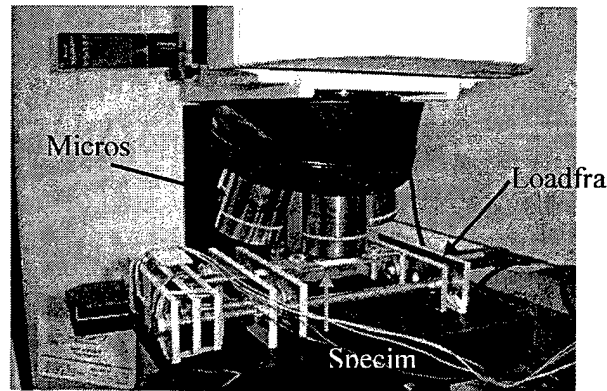


Figure 6 A small load frame for observing failure process in the composite

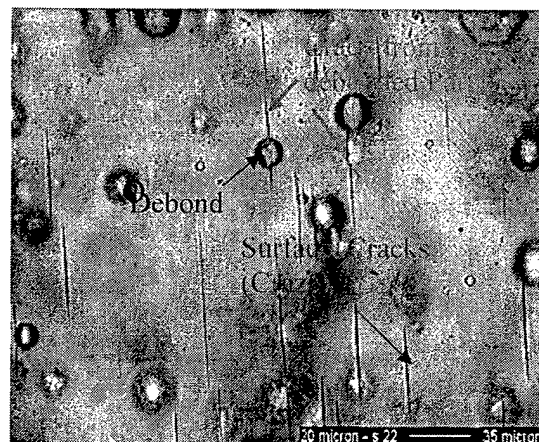


Figure 7 Observed failure modes in composite with 3 vol. % of 20  $\mu\text{m}$  aluminum oxide particles

#### Reference

1. Q.-G. Zeng and C.T. Sun, "Novel Design of a Bonded Lap Joint," *AIAA Journal*, Vol. 39, No. 10, October 2001, pp. 1991-1996.
2. C.T. Sun, A. Deo and H. Qian, "Effects of Shape and Surface Area of Fiber in Short Fiber Composites," 45<sup>th</sup> *AIAA/ASME/ASCE/AHS/ASC Structures, Structural Dynamics, and Materials (SDM) Conference*, Palm Springs, CA, April 19-22, 2004.

#### Personnel Supported

- A. Deo-- MS student
- J. Cho -- PhD student
- H. Qian--graduated with MS degree

#### **Publications**

- Deo and C.T. Sun, "Effect of Thickness of Platelet on Load Transfer Efficiency in Platelet-Reinforced Composites," *Proceedings of the 19<sup>th</sup> Technical Conference, American Society for Composites*, Atlanta, Georgia, October 18-20, 2004.
- J. Cho, "Failure Mechanisms in Polymer Composites Containing Micro and Nano Particles," *Proceedings of the 19<sup>th</sup> Technical Conference, American Society for Composites*, Atlanta, Georgia, October 18-20, 2004.

#### **New Discoveries, Inventions, or Patent Discoveries**

None

#### **Honors and Awards**

1993	Fellow, American Society of Mechanical Engineers.
1994	Fellow, American Institute of Aeronautics & Astronautics.
1995	American Society for Composites 1995 Distinguished Research Award
1996	Neil Armstrong Distinguished Professor of Aeronautical and Astronautical Engineering, Purdue University
1997	AIAA Structures, Structural Dynamics and Materials Award
1997	Medal of Excellence in Composite Materials, Center for Composite Materials, University of Delaware.
1998	Fellow, American Society for Composites
2004	Research Award for excellence in faculty research, Schools of Engineering, Purdue University.